

INTERNAL CORRESPONDENCE

C-49-03-92-

TO:

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DATE: MARCH 15, 1992

FROM:

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SUBJECT: CYANIDE OXIDATION TEST RESULTS FOR POND 207C

EG&G ROCKY FLATS SOLAR POND STABILIZATION PROJECT

REVISION NO. 1

1.0 PURPOSE

The characterization data for Pond 207C indicate that the total cyanide concentration in the water is above the LDR standard for wastewater and that the total cyanide concentration in the sludge is below the LDR standard for non-wastewater (see attached memo). The characterization data for Pond 207C waterS indicates that the total cyanide concentration ranges from 3.3 to 20 mg/l with an average concentration of 7.7 mg/l. The LDR standard for total cyanide is 1.2 mg/l for F006 listed wastes, which is applicable for Pond 207C.

Testing was conducted to oxidize the cyanide in Pond 207C using calcium hypochlorite, chlorine dioxide, and hydrogen peroxide. The testing was conducted initially on 207C slurry (5 parts water, 1 part crystal, and 1 part silty sludge), however; as the testing progressed, tests were conducted only on the water to conserve quantities of silty sludge and crystal.

1.1 ALKALINE CHLORINATION

The oxidation of cyanide with calcium hypochlorite and chlorine dioxide is commonly referred to as alkaline chlorination. Typically, this process is conducted in two steps. In the first step, CN is oxidized to cyanate, and in the second step, cyanate is oxidized to carbon dioxide and nitrogen. The first step is conducted at a pH of 9.0 to 10.0, and the second step is conducted at a pH of 8.0 to 8.5. In many cases, only oxidation to cyanate is required to achieve discharge limits.

The stoichiometric requirement for the oxidation of cyanide to cyanate is approximately 2.7 mg/l of chlorine per mg/l of CN . To oxidize cyanate to completion, 2.4 mg/l of chlorine per mg/l of is required. Therefore, a maximum chlorine dosage of approximately 119 mg/l should be sufficient to oxidize the maximum TO: TED BITTNER
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total cyanide to cyanate. The dosage of chlorine should be based on the total cyanide concentration because complexed cyanide should dissociate to maintain equilibrium as the free cyanide is oxidized.

1.1.1 Cyanide Oxidation With Calcium Hypochlorite

Tests were conducted to determine if chlorination of 207C composite waste using calcium hypochlorite would sufficiently oxidize the cyanide present in the pond, and if so, the optimal conditions. Calcium hypochlorite dosage, pH, and retention time were the parameters evaluated in the oxidation testing.

The experiments were designed to evaluate calcium hypochlorite concentrations from approximately 100 ppm to 10,000 ppm; varying the retention time from 30 minutes to 24 hours; and varying the pH from 9.0 to 11.0. Four tests were conducted to determine the effect of varying the above parameters. Each test is briefly described below:

- Test #1 This test was conducted on 1/09/92. The testing parameters were calcium hypochlorite concentration, pH, and retention time. The retention time was varied from 15 to 30 minutes, the calcium hypochlorite dosage was varied from 100 to 5000 ppm, and the pH was varied from 9.0 to 11.0.
- Test #2 This test was conducted on 1/10/92. This test used a 30 minute retention time, a pH of 9.0 & 10.0, and calcium hypochlorite concentrations of 100 ppm & 500 ppm.
- Test #3 This test was conducted on 1/14/92. This test used a 30 minute retention time with the pH varying from 9.0 to 10.0, and calcium hypochlorite concentrations of 5000 ppm and 10,000 ppm.
- Test #4 This test was conducted on 1/16/92. This test varied retention time with a constant pH and calcium hypochlorite concentrations of 1000 ppm and 5000 ppm.

1.1.2 Cyanide Oxidation With Chlorine Dioxide

The testing with calcium hypochlorite was unsuccessful in achieving the LDR standards for total cyanide. Because chlorine dioxide is a stronger oxidant than calcium hypochlorite, tests were conducted to determine if a stronger oxidant would oxidize the total cyanide. Testing with chlorine dioxide was conducted using a concentration of 1500 ppm at a pH of 9.7. This concentration is well above the calculated stoichiometric requirement for oxidation of all total cyanide. Samples were collected after 1 hour and 24 hours of reaction time.

1.2 HYDROGEN PEROXIDE OXIDATION

The tests using alkaline chlorination were unsuccessful in oxidizing the cyanide; therefore, hydrogen peroxide was tested to determine its ability to oxidize the cyanide. Oxidation with hydrogen peroxide is generally conducted at a pH of 8.5 to 10.0.

The cyanide is first oxidized to cyanate which is then slowly hydrolyzed to form carbon dioxide and ammonia. This reaction rate is greatly increased by traces of catalytic metals, such as copper and iron. Typically, 1.3 mg/l of hydrogen peroxide is required to oxidize 1.0 mg/l of cyanide.

Three separate tests were conducted using hydrogen peroxide. The testing evaluated hydrogen peroxide, hydrogen peroxide with cupric sulfate, and hydrogen peroxide with ferrous sulfate (Fenton's Reagent).

2.0 PROCEDURE

The initial testing for cyanide oxidation was conducted on the combined material slurried at a 1/1/5 ratio of silty sludge, crystal, and pond water, respectively. As testing proceeded and it was determined that more testing was needed because of the unsuccessful results, testing was conducted only on the water from Pond 207C. The decision to conduct tests on only water was made to avoid using large quantities of the slurry which was needed for testing of the stabilization formulas.

Prior to oxidation, total and amenable cyanide analyses were performed on the composited sludge or water and recorded as the baseline concentration. Typically, a baseline analysis was conducted at the beginning of each separate experiment. A summary of the baseline results are shown in Table 2-1.

The data in Table 2-1 indicate that there is significant variability in the cyanide analysis, which is likely caused by the cyanides being complexed with metals and/or the high concentration of dissolved solids in 207C water. The analysis for weak and dissociable cyanide (W&D) may be a more accurate measurement of amenable cyanide, which typically was reported as a negative number in the characterization report. Negative numbers are commonly associated with amenable cyanide analysis when cyanides are complexed with metals. The baseline average concentration for total cyanide is 5.4 mg/l.

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2.1 CYANIDE OXIDATION WITH CALCIUM HYPOCHLORITE

TEST 1

Three experiments were performed using the following concentrations of calcium hypochlorite: 100 ppm, 1000 ppm, and 5000 ppm. The calcium hypochlorite had a purity of 65 %. In each experiment, the pH was adjusted using hydrochloric acid or sodium hydroxide to achieve pH values of 9.0, 9.5, 10.0, 10.5, and 11.0. Each experiment was designed for sample collection for analysis after 15 and 30 minutes of retention time. After collecting the sample, the oxidation reaction was intended to be stopped by addition of sodium thiosulfate. To determine the amount of sodium thiosulfate to be added, residual chlorine was monitored by using a chlorine probe.

When the samples were submitted for analysis, the laboratory technician checked for residual chlorine using potassium iodide (KI) paper and found that all of the samples contained chlorine. Thus, the reactions had not been stopped after the intended reaction time. The decision was made to forego analysis because all the reaction times could not be determined (Note: at that time it was believed that the oxidation of cyanide would be relatively straight forward). Based on the observations of the technician it was realized that the chlorine probe was unable to provide accurate results because of the high dissolved solids concentrations. A determination was made to use both KI paper and ortho-toluidine to assure no residual chlorine would be present in samples generated in the future.

Because of the concern that the reaction time was not known, not all of the samples were analyzed. Three of the samples were analyzed when they were submitted and five samples were analyzed at a later date (approximately 11 days later) to determine if any oxidation occurred. These samples were analyzed after testing determined that the cyanide was not simple to oxidize and that a long reaction time might be required if the cyanides are complexed with metals. These results are provided in Section 3.0. Table 2-2 summarizes the trials conducted during Test 1.

TEST 2

In this experiment the retention time was held constant at 30 minutes by stopping the oxidation reaction with sodium thiosulfate. The 207C composite sludge pH and concentration of calcium hypochlorite were varied from a starting point of 10.3 to the desired pH using hydrochloric acid. The trials performed are as shown in Table 2-3. The results from the total and amenable cyanide analyses are provided in Section 3.0.

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TEST 3

In this experiment the retention time was held constant at 30 minutes while varying the pH and concentration of calcium hypochlorite. The initial pH of the 207C composite was 10.1. Hydrochloric acid was used to lower the pH to the desired level. The experiment trial runs are as shown in Table 2-4. The results from the total and amenable cyanide analysis are provided in Section 3.0.

TEST 4

This test used 207C pond water with an initial pH of 9.8, which was not adjusted. Two experiments were performed at calcium hypochlorite concentrations of 1000 ppm and 5000 ppm. The reaction times tested were 30 minutes, 90 minutes, 180 minutes, and 24 hours. The reaction was stopped using sodium thiosulfate. The samples were analyzed for total and amenable cyanide. The results are provided in Section 3.0. Table 2-5 summarizes the trials conducted during Text 4.

2.2 OXIDATION USING CHLORINE DIOXIDE

This experiment was conducted to determine the effectiveness of chlorine dioxide to oxidize the cyanide found in Pond 207C water. Retention times of 1 hour and 24 hours were tested with all other variables held constant. The 207C pond water had an initial pH of 10.2.

Because of the difficulties in achieving reproducible results, three analytical testing methods were used to determine the concentration of cyanide present. The methods are: ASTM D2036 Total and Amenable Cyanide Method, EPA 335.2 Total and Amenable Cyanide Method, and ASTM D2036 Weak & Dissociable Cyanide Method. The 207C pond water was submitted for baseline analysis using all three testing procedures. The results are shown in Table 2-1.

A saturated solution of chlorine dioxide was prepared at a concentration of 3000 ppm. The chlorine dioxide solution (600 ml) was added to 600 ml of 207C water to obtain a concentration of 1500 ppm chlorine dioxide. The pH of the mixture dropped from 10.2 to 9.3 when the chlorine dioxide solution was added to the pond water. To increase the pH, 42 ml of 1N sodium hydroxide was added which increased the pH to 9.7. After the addition of the sodium hydroxide the solution became very cloudy and a significant amount of fine white precipitate was formed. Samples were submitted after 1 hour and 24 hours of contact time with constant mixing. The results are provided in Section 3.0.

2.3 CYANIDE OXIDATION WITH HYDROGEN PEROXIDE

Three tests were conducted to evaluate hydrogen peroxide for oxidizing cyanide in Pond 207C water. The first test used hydrogen peroxide at a concentration of 1000 ppm. The second experiment evaluated hydrogen peroxide with cupric sulfate to enhance the reaction. The third experiment used hydrogen peroxide and ferrous sulfate at a pH of 5.0. The third experiment is representative of Fenton's Reagent, which is a strong oxidant.

TEST 1

Six (6) ml of 50% hydrogen peroxide was added to 3 liters of 207C water to obtain a concentration of 1000 ppm of hydrogen peroxide. Samples were collected after 1 hour and 24 hours of reaction time. The results of this testing are provided in Section 3.0.

TEST 2

Four liters of 207C Pond Water were collected and a 1-liter aliquot was submitted for baseline cyanide analysis using ASTM D2036 by colorimetric and titration analysis for both total and amenable cyanide. The results are provided in Section 3.0.

The remaining 3 liters of sample were divided into 2 liter and 1 liter aliquots which were used for Tests 2 and 3, respectively. The pH of the 2-liter sample was adjusted to 10.0 with 12 grams of hydrated lime. Hydrogen peroxide (50%) was added at a concentration of 2000 ppm (8.0 ml) and cupric sulfate was added at a concentration of 50 ppm (100 mg). The mixture was stirred continuously and samples were removed after 1 hour and 24 hours. The samples were submitted for cyanide analysis using ASTM D2036 colorimetric and titration method for both total and amenable cyanide. The results are provided in Section 3.0.

TEST 3

This test evaluated the use of Fenton's Reagent, a powerful oxidant, to destroy the cyanide in the 207C Pond water. Fenton's chemistry occurs at a pH of 5.0 when hydrogen peroxide is mixed with ferrous sulfate. The pH was adjusted by adding 19 ml of sulfuric acid to the 1 liter sample of 207C pond water. Hydrogen peroxide (50% solution) was added to achieve a concentration of 2000 ppm (4 ml) and ferrous sulfate was added to a concentration of 150 ppm (150 mg). The mixture was stirred continuously and samples were collected after 1 and 24 hours. The samples were submitted for total and amenable cyanide analysis. The results are provided in Section 3.0.

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3.0 RESULTS

The results from the cyanide oxidation tests are provided in the following section. The results from the cyanide analysis indicate that the sample matrix causes interferences with the analytical procedure. The data reported from the laboratory commonly have notes on those samples which were used for matrix spikes indicating that there are matrix interferences.

3.1 CYANIDE OXIDATION RESULTS USING CALCIUM HYPOCHLORITE

TEST 1

The results for Test 1 are shown in Table 3-1. These results are somewhat ambiguous because the oxidation reaction was not stopped at the proper time because of interferences with the chlorine Although the exact reaction time for each test is not known, it is greater than 24 hours in all cases. The results indicate that the LDR standards could not be achieved under the conditions that were tested. The dosages of calcium hypochlorite were above the required stoichiometric concentration to oxidize the cyanide under ideal conditions. Review of the data in Table 3-1 suggests that a reduction in the cyanide concentration occurred when compared to the average baseline concentration. However, since there was no baseline analysis performed at the time the samples were submitted, an exact determination of the destruction of cyanide can not be made. When the remaining tests are reviewed it will become apparent that the cyanide is not oxidized and differences in the cyanide concentrations are likely a result of analytical variances.

TEST 2

Test 2 was conducted to repeat the oxidation testing performed in Test 1. The initial testing was duplicated because of concerns in Test 1 regarding the inability to stop the cyanide oxidation. The results, provided in Table 3-2, suggest that as much as 70% of the total cyanide was reduced, although the cyanide was still above the LDR standard. However, there is also considerable variance in the final cyanide analytical data.

TEST 3

This experiment was conducted at high concentrations of calcium hypochlorite to ensure that the oxidizing agent was not the limiting factor. The results of this testing are shown in Table 3-3. The data does not indicate any significant reduction in cyanide concentrations and demonstrates the variability in the

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analytical results. The concentrations of cyanide were not reduced to levels below the LDR standards.

TEST 4

This test was conducted at a high concentration of calcium hypochlorite with longer retention times. The longer retention time was thought to be necessary if the cyanide was complexed with metals. The results are provided in Table 3-4. The data indicate that the analytical results are extremely variable. Samples collected after 30 and 90 minutes, at a concentration of 1000 ppm, suggest that the standard is achieved; however the same sample collected after 180 minutes and 24 hours indicate that the cyanide concentration increased from the samples collected earlier. The results that were below the LDR standards are likely analytical anomalies.

3.2 CYANIDE OXIDATION RESULTS WITH CHLORINE DIOXIDE

Chlorine dioxide was tried with the hope that a stronger oxidant would be more effective oxidizing the cyanide. Because of the 2:1 dilution after the addition of the chlorine dioxide solution, the measured cyanide concentration should be multiplied by 2 to correct for the dilution. The results are provided in Table 3-5. The results indicate that 1500 ppm chlorine dioxide with a maximum contact time of one day was unable to oxidize the cyanide to achieve the LDR standard.

Also shown in Table 3-5 are analytical results for cyanide using several different methods. Cyanide analysis using different methods was conducted to determine if a particular method would provide accurate results for amenable cyanide, which consistently produced negative values. The results demonstrate significant variability between the ASTM method and the EPA method. The weak and dissociable analysis provides a result which should be somewhat representative of amenable cyanide.

3.3 CYANIDE OXIDATION RESULTS WITH HYDROGEN PEROXIDE

Hydrogen peroxide was tested to determine if constituents in 207C pond water were interfering with the alkaline chlorination reaction. Three tests were conducted with hydrogen peroxide. The results are discussed below.

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TEST 1

Test 1 used hydrogen peroxide at a concentration of 1000 ppm, which is significantly higher than the stoichiometric requirement of 26 mg/l (maximum based on 20 mg/l of cyanide). The results shown in Table 3-6 indicate that hydrogen peroxide at 1000 ppm was unable to oxidize cyanide to achieve the LDR standard over a 24-hour reaction time.

Additionally, the hydrogen peroxide was unable to oxidize the weak and dissociable cyanide. The weak and dissociable cyanide should be readily oxidized with hydrogen peroxide. This observation suggests that the high salt content of the pond water interferes with the cyanide oxidation reaction.

Table 3-6 also indicates that there was significant variability in the analysis for cyanide. The results using the EPA method and the ASTM method do not correlate well.

TEST 2

Test 2 evaluated hydrogen peroxide with cupric sulfate. The addition of cupric sulfate helps to catalyze the cyanide oxidation reaction. As in Test 1, the dosage of hydrogen peroxide is well above stoichiometric requirements, as is the dosage of cupric sulfate. The results, shown in Table 3-7 indicate that the cyanide was not oxidized to levels below the LDR standards.

Table 3-7 also provides results of cyanide analysis by colorimetric and titration (ASTM D2036). These results also demonstrate significant variability.

TEST 3

Test 3 evaluated the effect of hydrogen peroxide with ferrous sulfate, which may enhance the oxidation of cyanide at a pH of 5.0. The results of this testing, provided in Table 3-8, indicate that cyanide was not oxidized to a level below the LDR standards. As in Table 3-7, there is significant variability between the colorimetric and titration analysis methods.

4.0 CONCLUSIONS

The analytical results for total cyanide clearly indicate that the 207C Pond water is difficult to analyze due to matrix interferences which are likely attributed to the high salt content. Analysis for amenable cyanide can not accurately be conducted based on the frequency of negative values that are reported. This observation suggests that much of the cyanide is complexed with metals.

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The cyanide present in Pond 207C water is extremely resistant to oxidation. Typically, alkaline chlorination is successful in reducing the concentration of cyanide; however, the testing conducted on the 207C waters did not significantly reduce the cyanide levels. Oxidation with hydrogen peroxide was also unsuccessful in reducing the levels of cyanide to below the LDR standards. Fenton's reagent, which is a very strong oxidant, was also unable to significantly reduce the cyanide concentration. Although there was significant variability in the analytical results, the data suggests that the oxidizing agents had almost no effect on the cyanide concentration. The reason for this observation is not clear, although it is likely related to the high salt content in the pond and/or the cyanide being complexed with metals.

TABLE 2-1
BASELINE ANALYSIS FOR CYANIDE

| MATRIX | METHOD | PROCEDURE | TOTAL CYANIDE (ppm) | AMENABLE CYANIDE (ppm) |
|--------|-----------|--------------|------------------------|------------------------------|
| Sludge | ASTM 2036 | COLORIMETRIC | 6.7 | -7.2 |
| Sludge | ASTM 2036 | COLORIMETRIC | 3.1 | -8.7 |
| Water | ASTM 2036 | COLORIMETRIC | 1.5 | -29 |
| Water | ASTM 2036 | COLORIMETRIC | 4.1 | -130 |
| Water | ASTM 2036 | COLORIMETRIC | 6.3 | 1.1 |
| Water | ASTM 2036 | TITRATION | 5.2 | < 5 |
| Water | ASTM 2036 | COLORIMETRIC | | 2.9(1) |
| Water | EPA 335.2 | COLORIMETRIC | 11 | -140 |

(1) This represents the analytical result for a weak and dissociable cyanide which should be similar to the value for amenable cyanide. This test was conducted because of the difficulties in analyzing the waste for amenable cyanide as shown by the negative results.

TABLE 2-2

CALCIUM HYPOCHLORITE OXIDATION TEST #1

| Trial # | Volume of 207C | рН | HCL Added | NaOH Added | Ca (C10) ₂ ppm | Ca(C10) ₂ Added |
|---------|-------------------|------|--------------|---------------|------------------------------|-------------------------------|
| 1 | 600 mi | 9.0 | 40 ml | | 100 | .06 g |
| 2 | 600 mi | 9.5 | 18.5 ml | | 100 | .06 g |
| 3 | 600 ml | 11.0 | _ | 38.5 ml | 1000 | .6 g |
| 4 | 600 ml | 9.0 | 38.5 ml | | 5000 | 3 g |
| 5 | 600 ml | 9.5 | 22 ml | | 5000 | 3 g |
| 6 | 600 mt | 10.0 | 7.0 ml | | 5000 | 3 g |
| 7 | 600 ml | 10.5 | | 9 ml | 5000 | 3 g |
| 8 | 600 ml | 11.0 | | 33 ml | 5000 | 3 g |

TABLE 2-3

CALDIUM HYPOCHLORITE OXIDATION TEST #2

| TRIAL # | 207C VOLUME | рН | HCL ADDED | Ca(CiO) ₂ ppm | Ca(CIO)₂ ADDED |
|------------|----------------|------|--------------|-----------------------------|-------------------|
| 1 | 600 ml | 9.0 | 38 ml | 100 mg/l | 0.06 g |
| 2 | 600 ml | 10.0 | 5 ml | 100 mg/l | 0.06 g |
| 3 | 600 ml | 9.0 | 39 mi | 500 mg/l | 0.30 g |
| 4 | 600 ml | 10.0 | 6 mi | 500 mg/l | 0.30 g |

TABLE 2-4

CALCIUM HYPOCHLORITE OXIDATION TEST #3

| TRIAL # | VOLUME 207C | рН | HCL ADDED | Ca(CIO) ₂ ppm | Ca(CIO) ₂ ADDED |
|------------|----------------|------|--------------|-----------------------------|-------------------------------|
| 1 | 600 ml | 9.0 | 42.5 ml | 5,000 | 3 g |
| 2 | 600 mi | 9.5 | 17.5 ml | 10,000 | 6 g |
| 3 | 600 mi | 10.0 | 2.5 ml | 5,000 | 3 g |
| 4 | 600 ml | 9.0 | 48.5 ml | 10,000 | 6 g |
| 5 | 600 mi | 9.5 | 19.5 ml | 5,000 | 3 g |
| 6 | 600 ml | 10.0 | 4.0 mi | 10,000 | 6 g |

TABLE 2-5

CALCIUM HYPOCHLORITE OXIDATION TEXT #4

| Trial # | Volume of 207C | рН | Ca(C10) ₂ ppm | Ca(C10) ₂ Added |
|---------|-------------------|-----|-----------------------------|-------------------------------|
| 1 | 1 200 ml | 9.8 | 1000 | 1.2 g |
| 2 | 1200 mi | 9.8 | 1000 | 1.2 g |
| 3 | 1200 ml | 9.8 | 1000 | 1.2 g |
| 4 | 1 200 ml | 9.8 | 1000 | 1.2 g |
| 5 | 1200 ml | 9.8 | 5000 | 6 g |
| 6 | 1 200 ml | 9.8 | 5000 | 6 g |
| 7 | 1200 ml | 9.8 | 5000 | 6 g |
| 8 | 1200 mi | 9.8 | 5000 | 6 g |

TABLE 3-1

CALCIUM HYPOCHLORITE OXIDATION RESULTS, TEST #1

| TRIAL # | Ca(CIO)2 (ppm) | RET. TIME | рН | TOTAL CN | AMEN. CN |
|---------------------|-------------------|--------------|------|-------------|-------------|
| LDR Limit | | | | 1.2 | 0.1 |
| Average Baseline | NA | NA | NA | 5.4 | |
| 1 | 100 | UNKNOWN | 9.0 | 1.3 | NR |
| 2 | 100 | UNKNOWN | 9.5 | 2.6 | NR |
| 3 | 1000 | UNKNOWN | 11.0 | 1.9 | NR |
| 4 | 5000 | UNKNOWN | 9.0 | 2.8 | NR |
| 5 | 5000 | UNKNOWN | 9.5 | 2.9 | NR |
| 6 | 5000 | UNKNOWN | 10.0 | 1.8 | NR |
| 7 | 5000 | UNKNOWN | 10.5 | 1.8 | NR |
| 8 | 5000 | UNKNOWN | 11.0 | 1.9 | NR |

NR - no results, test not performed.

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TABLE 3-2

CALCIUM HYPOCHLORITE OXIDATION RESULTS, TEST #2

| TRIAL # | Ca(CIO) ₂ (ppm) | RET. TIME | рН | TOTAL CN | AMEN. CN |
|---------------------|-------------------------------|--------------|------|-------------|-------------|
| LDR Limit | | | | 1.2 | 0.1 |
| Baseline Test #2 | NA | NA | 10.1 | 6.7 | -7.2 |
| 1 | 100 | 30 min. | 9.0 | 3.2 | 0.69 |
| 2 | 100 | 30 min. | 10.0 | 3.9 | 0.97 |
| 3 | 500 | 30 min. | 9.0 | 2.0 | -2.2 |
| 4 | 500 | 30 min. | 10.0 | 2.7 | 0.82 |

NR - no results, test not performed.

TABLE 3-3

CALCIUM HYPOCHLORITE OXIDATION RESULTS TEST #3

| TRIAL # | Ca(ClO)2 (ppm) | RET. TIME | рН | TOTAL CN | AMEN. CN |
|---------------------|-------------------|--------------|------|-------------|-------------|
| LDR Limit | | | | 1.2 | 0.1 |
| Baseline Test #3 | NA . | NA | 10.3 | 3.1 | -8.7 |
| 11 | 5000 | 30 min. | 9.0 | 4.5 | -9.2 |
| 2 | 10,000 | 30 min. | 9.5 | 3.8 | -0.7 |
| 3 | 5000 | 30 min. | 10.0 | 3.2 | -7.7 |
| 4 | 10,000 | 30 min. | 9.0 | 4.6 | 1.3 |
| 5 | 5000 | 30 min. | 9.5 | 3.6 | -9.4 |
| 6 | 10,000 | 30 min. | 10.0 | 1.7 | -19 |

NA - not applicable

NR - no results, test not performed.

TABLE 3-4

CALCIUM HYPOCHLORITE OXIDATION RESULTS, TEST #4

| TRIAL # | Ca(CIO) ₂ (ppm) | RET. TIME | рН | TOTAL CN | AMEN. CN |
|---------------------|-------------------------------|--------------|-----|-------------|-------------|
| LDR Limit | | | | 1.6 | 0.1 |
| Baseline Test #4 | NA | NA | 9.8 | 1.5 | -29 |
| 1 | 1000 | 30 min. | 9.8 | 1.1 | 0.063 |
| 2 | 1000 | 90 min. | 9.8 | 0.97 | -3.9 |
| 3 | 1000 | 180 min. | 9.8 | 1.7 | -1.9 |
| 4 | 1000 | 24 hrs. | 9.8 | 2.0 | -4.4 |
| 5 | 5000 | 30 min. | 9.8 | 1.7 | 0.95 |
| 6 | 5000 | 90 min. | 9.8 | 1.8 | -1.2 |
| 7 | 5000 | 180 min. | 9.8 | 1.6 | -4.9 |
| 8 | 5000 | 24 hrs. | 9.8 | 1.4 | -14 |

NR - no results, test not performed.

TABLE 3-5
CHLORINE DIOXIDE OXIDATION RESULTS

| OXIDANT RET. TIME | i | | CONC. | | Cyanide by ASTM D2036 | | nide by 335.2 | Cyanide by ASTM D2036 |
|----------------------|---------|-------|-------|---------------------------|--------------------------|-------|------------------|--------------------------|
| | TIME | (ppm) | pН | Total | Amen. | Total | Amen. | Weak & Diss. |
| LDR Limit | | | | 1.2 | 0.1 | 1.2 | 0.1 | _ |
| Baseline | 0 | N/A | 10.2 | 4.1 | -130 | 11 | -140 | 2.9 |
| C10 ₂ | 1 hr | 1500 | 9.7 | 1.8 (3.6) ¹ | -99 | N/A | N/A | N/A |
| C102 | 24 hrs. | 1500 | 9.7 | 2.6 (5.2) ¹ | -11 | N/A | N/A | N/A |

¹ Concentration of sample should be multiplied by 2 because of the dilution from Chlorine Dioxide solution.

TABLE 3-6
HYDROGEN PEROXIDE OXIDATION RESULTS, TEST #1

| OXIDANT | RET. TIME | CONC. | рН | TOTAL & AMENABLE | AMENABLE EPA 335.2 | | CYANIDE BY ASTM D2036 |
|-------------------------------|--------------|-------|------|--------------------------|--------------------|-------|--------------------------|
| | | | | CYANIDE BY ASTM D2036 | TOTAL | AMEN. | WEAK & DISS. |
| LDR Limit | *** | | | 1.2/0.1 | 1.2 | 0.1 | |
| Base- line | 0 | NA | 10.2 | 4.1 /-130 | 11 | -140 | 2.9 |
| H ₂ O ₂ | 1 hr. | 1000 | 10.1 | 0.16 /-93 | NA | NA | NA |
| H ₂ O ₂ | 24 hrs. | 1000 | 10.1 | 4.3 /-11 | 5.9 | -140 | 2.4 |

TABLE 3-7
HYDROGEN PEROXIDE OXIDATION RESULTS TEST #2

| OXIDANT | RET. TIME | CONC. OF H ₂ O ₂ AND CuSO ₄ | рН | CYANIDE BY ASTM D2036 COLORIMETRIC | | CYANIDE BY ASTM D2036 TITRATION | |
|--|--------------|--|-----|--|-------|---------------------------------------|-------|
| | | (ppm) | | TOTAL | AMEN. | TOTAL | AMEN. |
| LDR Limit | | | | 1.2 | 0.1 | 1.2 | 0.1 |
| Baseline | 0 | NA | 9.2 | 6.3 | 1.1 | 5.2 | < 5 |
| H ₂ O ₂ & CuSO ₄ | 1 HR. | 2000/50 | 10 | 5.7 | 2.5 | 16 | < 5 |
| H ₂ O ₂ & CuSO ₄ | 24 HRS. | 2000/50 | 10 | 5.3 | 0.62 | 5.2 | < 5 |

NA - not applicable

NR - no results, Test not performed

TABLE 3-8 HYDROGEN PEROXIDE OXIDATION RESULTS TEST #3

| OXIDANT | RET. TIME | CONC. OF H ₂ O ₂ AND CuSO ₄ (ppm) | pН | CYANIDE BY ASTM D2036 COLORIMETRIC | | CYANIDE BY ASTM D2036 TITRATION | |
|--|-----------|--|-----|--|-------|---------------------------------------|-------|
| | | | | TOTAL | AMEN. | TOTAL | AMEN. |
| Base line | 0 | NA | 9.2 | 6.3 | 1.1 | 5.2 | < 5 |
| LDR Limit | | | | 1.2 | 0.1 | 1.2 | 0.1 |
| H ₂ O ₂ & FeSO₄ | 1 HR. | 2000/150 | 5.0 | 4 | 5.9 | 10 | -5.2 |
| H₂O₂ & FeSO₄ | 24 HRS. | 2000/150 | 5.0 | 3.3 | -390 | 16 | -340 |

NA - not applicable NR - no results, test not performed